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METHOD 375.2

I. SCOPE AND APPLICATION:

This is a sample collection procedure designed for the analytical determination of sulfate in water using automated colorimetry. This automated method is applicable to drinking, ground, and surface water as well as domestic and industrial wastewater. The application range is 3 to 300 mg SO₄/liter. The sensitivity of the method can be increased by a minor modification to analyze samples in the range of 0.5 to 30mg SO₄/liter. Approximately 30 samples per hour can be analyzed.

<u>Analyte</u>	<u>Chemical Abstract Services Registry Numbers (CASRN)</u>
Sulfates (SO ₄)	not applicable

II. REAGENTS: none

III. MATERIALS:

- 500 ml or 1-liter amber borosilicate sample bottle fitted with screw caps lined with TFE-fluorocarbon.
- Latex gloves
- Protective eyewear
- Folding card table
- Paper towels
- Kim wipes
- Pliers

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything which may fall into the sample container.
2. Open the tap and allow the system to flush for about 10 minutes. This should be adequate to allow the water temperature to stabilize and get a representative sample.
3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.
4. Remove the cap from the sample container.
5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.
6. Remove the bottle from the flow and recap. Dry the sample container and affix the sample label to the bottle. Collect samples in duplicate if necessary.

V. SAMPLE TRANSPORT:

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each sample bottle. Place the sample bottle into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use “blue ice” as the voa samples will never adequately chill. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. DEFINITIONS:

- A. *Aliquot*: A measured portion of a sample taken for analysis.
- B. *Matrix effects*: The influence of the sample matrix or sample components upon the ability of analytical methods to qualitatively identify and quantitatively measure target compounds in environmental samples

VII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

VIII. SUMMARY OF METHOD:

METHOD 375.2: The sample is first passed through a sodium cation-exchange column to remove multivalent metal ions. The sample sulfate-containing sample is then reacted with an alcohol solution of barium chloride and methylthymol blue (MTB) at a pH of 2.5-3.0 to form barium sulfate. The combined solution is raised to a pH of 12.5-13.0 so that excess barium reacts with MTB. The uncomplexed MTB color is grey. If the MTB is all chelated with barium, its color is blue. Initially, the barium and MTB are equimolar and equivalent to 300 mg of SO₄/liter; thus, the amount of uncomplexed MTB is equal to the amount of sulfate present.